Kimball Physics, Inc. Technical Bulletin # LaB<sub>6</sub>- 06B August 1991 P.B. Sewell

## KIMBALL PHYSICS ES-423E LaB<sub>8</sub> CATHODE OPERATING INSTRUCTIONS FOR LEICA/CAMBRIDGE STEREOSCAN SERIES SEM's

#### Introduction

The Style 90-20, ES-423E LaB<sub>6</sub> Cathode is designed to be used in scanning electron microscopes that normally operate with a high emission current in the range of  $60 - 100 \ \mu$ A. The 90° cone at the emitting end of the cathode is terminated with a 20  $\mu$ m diameter flat, parallel to the low work function <100> surface of the LaB<sub>6</sub> single crystal.

It is desirable that the reader be familiar with the information in Kimball Physics Technical Bulletin  $#LaB_6-01$ , "General Guidelines for Operating ES-423E LaB<sub>6</sub> Cathodes," and the Leica "LaB<sub>6</sub> Option Operating Instructions" in order to better understand the material presented here.

In the saturated emission mode, when emission is limited to the area of the <100> flat, the operating temperature of the cathode is in a range where long life can be achieved in suitable gun vacuum. A small cross-over of about 13  $\mu$ m diameter is produced and there is little sacrifice in axial brightness as compared to other tip configurations.

In the Leica/Cambridge series of SEM's, with good gun vacuum in the range of  $1 - 2 \times 10^{-7}$  torr, cathode lifetimes in excess of 2000 hours can be expected.

#### Cathode Characteristics

The ES-423E Cathode is based on a carbon resistance heater that has a room temperature resistance of about 2.1 ohms and an operating resistance of about 1.3 ohm. The resistance characteristics are shown in Figure 1 together with those of a typical refractory-metal based cathode with a positive temperature coefficient. With some power supplies, the difference in behavior of the cathode resistance as a function of temperature, may result in abnormal behavior during initial heating of the cathode.

<u>NOTE</u>: With Leica/Cambridge instruments, the high starting resistance of the ES-423E Cathode may cause the REPLACE CATHODE lamp to remain on, as the cathode is first heated. As the temperature is further increased, and cathode resistance drops, this lamp should go out.



Figure 1. Resistance characteristics of an ES-423E Cathode as compared to a typical wire cathode such as a tungsten or a Denka M3 cathode.

Table 1 lists the temperature as a function of the cathode current in the near-linear region of the operating curve normally used in scanning electron microscopy. The cathode temperature characteristics are shown in Figures 2 and 3. While the cathode can be heated to 2150 K (2.6 A) without immediate failure, continuous operation above 2.0 A will reduce the lifetime of the cathode to hundreds of hours rather than thousands of hours.

### Installation and Initial Operation

The cathode should be set at a height of  $125 \ \mu m$  or at the value specified by the Leica/Cambridge Service Engineer using an LaB<sub>6</sub> Wehnelt 1.0 mm diameter aperture. Always use a new or carefully cleaned aperture with a new LaB<sub>6</sub> cathode. Be particularly careful to avoid leaving any particulate insulating material in the region of the Wehnelt aperture.

Also check the condition of the edge of the Wehnelt aperture facing the cathode. This should be free of any sharp protrusions or burrs, and if needed, should be polished with diamond paste to ensure a smooth rounded edge. Sharp corners on the aperture may cause arcing between the Wehnelt and the cathode, resulting in emission instability and even shut-down of the instrument.

### TABLE 1

#### Temperature Calibration for ES-423E Cathode

Data taken from experimental calibration curves over the range of 1500 K to 2200 K. Using an approximation of the linear relationship over the range of 1750 K to 1950 K.

$$T = 1740 + (I_t - 1.8) \ge 500,$$

where  $I_t$  is the measured cathode current. Estimated accuracy over this range is about  $\pm 20$  K.

Temp	Current	Temp	Current	Temp	Current
IKI	[Amps]	IKI	[Amps]	IKI	[Amps]
1750	1.82	1850	2.02	1950	2.22
1755	1.83	1855	2.03	1955	2.23
1760	1.84	1860	2.04	1960	2.24
1765	1.85	1865	2.05	1965	2.25
1770	1.86	1870	2.06	1970	2.26
1775	1.87	1875	2.07	1975	2.27
1780	1.88	1880	2.08	1980	2.28
1785	1.89	1885	2.09	1985	2.29
1790	1.9	1890	2.1	1990	2.3
1795	1.91	1895	2.11	1995	2.31
1800	1.92	1900	2.12	2000	2.32
1805	1.93	1905	2.13	2005	2.33
1810	1.94	1910	2.14	2010	2.34
1815	1.95	1915	2.15	2015	2.35
1820	1.96	1920	2.16	2020	2.36
1825	1.97	1925	2.17	2025	2.37
1830	1.98	1930	2.18	2030	2.38
1835	1.99	1935	2.19	2035	2.39
1840	2.0	1940	2.2	2040	2.4
1845	2.01	1945	2.21	2045	2.41

<u>CAUTION</u>: When first using the ES-423E Cathode in an instrument with the AUTO RUN-UP feature, make the initial run-up manually. If the instrument has been programmed for a cathode with higher operating current, the auto run-up could damage the ES-423E Cathode. Increase the temperature of the cathode manually and when the final operating condition is determined, save this setting for future use.

After evacuation of the instrument, follow the start-up procedure as recommended: With the filament OFF, slowly raise the accelerating voltage to the maximum value to initiate any discharges between the Wehnelt and the Anode due to contamination. When the high voltage is stable, reduce the accelerating voltage to zero and begin the recommended start-up procedure.

<u>NOTE</u>: If discharges occur between the Wehnelt and the Anode while there is electron emission, then ions produced by the pressure pulse associated with the discharge will be accelerated back to the tip of the cathode and may physically damage and chemically contaminate the emitting area of the cathode tip. Emission may or may not recover after a discharge of this type.



Figure 2. Temperature versus cathode current for the ES-423E Cathode on typical SEM and TEM cathode bases. Straight line approximation (same data as Table 1) is accurate to about ± 20 K.

During the initial operation in a recently air-exposed electron gun, raise the cathode temperature and accelerating voltage at a rate which maintains the gun pressure always below  $10^{-6}$  torr. After the outgassing of the gun assembly and the achievement of operating pressures in the range of 1 to 2 x  $10^{-7}$  torr, the cathode can be switched ON and OFF rapidly, without suffering from thermal shock damage.

# Setting Emission

At the selected kV, watch the Emission Pattern as the temperature of the cathode is raised. The temperature should be increased until the first peak of axial intensity is achieved. The EMP should appear as shown in Figure 4a, being an intense, narrow, near-Gaussian central peak in the center of a weak cross. As seen in Figure 5, this is observed experimentally at a temperature of about 1780 K or a cathode current of about 1.88 A.

This operating point gives a high axial brightness and excellent resolution. However, here the emission is temperature limited so the specimen current is sensitive to changes in the cathode temperature (power supply fluctuations and contact resistance instabilities) and work function changes due to gas adsorption on the <100> flat.

NOTE: During the initial operation of a new cathode, gas evolution from the liner tube region of the microscope can result in deactivation of the tip of the cathode for short periods of time. Oscillations of the EMP can result in Leica/Cambridge instruments, due to the nature of the bias control circuit. These oscillations may occur as the total power in the liner tube changes, e.g., when changing from say 20 kV to 40 kV. These initial instabilities will disappear as the liner tube is cleaned of adsorbed gases. Some instabilities of this type may occur during the first day of operation after loading a new cathode or changing a liner tube or beam apertures.

If banding appears in photographic recording and is due to emission instability on the first peak position, then the temperature of the cathode should be increased to the full saturation position, Figure 4b as seen in the EMP. Here the emission is limited to the <100> surface of the cathode and is space charge limited. The EMP is a slightly broader Gaussian distribution and has slightly reduced axial intensity.

Operating in the full saturation mode can reduce the effects of cathode temperature fluctuations and gas deactivation. In this more stable mode, the operating temperature is about 1820 to 1830 K, corresponding to a cathode current of about 1.95 to 2.0 A.

The low-field and high-field temperature estimates of Figure 5 are the calculated operating temperatures for 80  $\mu$ A of emission from a Style 90-20 cathode, assuming that at saturation, all the emission comes uniformly from the 20  $\mu$ m diameter <100> surface truncation. The low-field estimate is based on a zero-field work function for the LaB<sub>6</sub> <100> surface of 2.69 eV and the high-field estimate based on a field reduced work function of 2.6 eV. This latter condition is found experimentally in triode guns operating with an average gun field of about 3 - 5 kV/mm.

### Life of Cathodes

The life of the cathode is generally determined by the vacuum conditions in the gun as described in Kimball Physics Technical Bulletin #LaB<sub>6</sub>-02, "The Relationship Between LaB<sub>6</sub> and Gun Vacuum."



Figure 3. General characteristics of the ES-423E LaB6 Cathode.



Figure 4a. EMP (EMission Pattern) in a Leica 360 SEM at first peak for an ES-423E LaB6 Cathode, Style 90-20.



Figure 4b. EMP in a Leica 360 SEM at full saturation for an ES-423E LaB6 Cathode, Style 90-20.



Figure 5. Experimentally observed operating conditions for an ES-423E Cathode, Style 90-20, in a Leica 360 at a height setting of 0.125 mm in an LaB<sub>6</sub> Wehnelt.

For operating currents below about 2.0 A the cathode life, as determined by the evaporation of the  $LaB_6$  crystal, is measured in thousands of hours. However, with an increase in cathode current, the evaporation of the carbon legs may become the life limiting factor for the cathode. At a current of 2.15 A, the life has been reduced by a factor of about 10 from the life at 2.0 A.

Higher cathode temperatures are generally required at low operating voltages, particularly if the Wehnelt to Anode spacing has not been adjusted to compensate for the lower gun field. Hence, shorter life might be expected in some operating conditions at low kV.

Over-saturation can also result in excess cathode operating temperatures. The operator should be sure to adjust for saturation so that the minimum cathode current is used at this setting. To ensure long cathode life, avoid operating the cathode with a current in excess of 2.0 A for extended periods of time.

Additional details are available in the following Kimball Physics Technical Bulletins:

- LaB<sub>6</sub>-01: General Guidelines for Operating ES-423E LaB<sub>6</sub> Cathodes
- LaB<sub>6</sub>-02: The Relationship Between LaB<sub>6</sub> and Gun Vacuum
- LaB<sub>6</sub>-03: Emission Drift LaB<sub>6</sub> Gun Stability
- LaB<sub>6</sub>-04: Oxygen Activation of LaB<sub>6</sub> Cathodes -The Double Saturation Effect
- LaB<sub>6</sub>-05: Kimball Physics ES-423E LaB<sub>6</sub> Cathode Style 60-06 (60° Included Cone Angle, 6μm Diameter Flat)
- LaB<sub>6</sub>-07: Recovery of Emission from ES-423E LaB<sub>6</sub> Cathodes Following a Vacuum Dump